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Smart Concrete for Enhanced Nondestructive Evaluation

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Abstract
The authors recently investigated the use of conductive concrete to enhance nondestructive evaluation (NDE) capa- bilities. Preliminary results have shown that a conductive concrete can facilitate the utilization of an eddy current technique, where damages in a conductive specimen were easier to detect compared with a non-conductive substrate. While such results demonstrated the promise of using conductive concrete to facilitate and potentially accelerate the NDE process, the fabrication of an homogeneous conductive concrete is technically or economically challenging, depending on the conductive filler used in the process. In this paper, we propose a new cementitious composite to accelerate NDE. The composite uses inexpensive carbon black particles (CB) and a block-copolymer. The purpose of the block co-polymer, a styrene-ethylene-butylene-styrene (SEBS), is to facilitate the creation of conductive chains, therefore reducing the necessary concentration of conductive filler required to achieve electrical percolation. Several cementitious composite specimens of various concentrations of CB are fabricated, and results show that the utilization of SEBS reduces the electrical percolation threshold by approximately 50% with a gain on electrical conductivity relative to a non-conductive specimen mix of approximately 33%. Strain-sensing tests also demonstrate that SEBS-based specimens have good sensing properties, but lag behind those of conductive concrete specimens fabricated with CB only.

Keywords
nondestructive evaluation, eddy current, conductive concrete, smart concrete, structural health monitoring, carbon black, SEBS

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Smart Concrete for Enhanced Nondestructive Evaluation

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ABSTRACT

The authors recently investigated the use of conductive concrete to enhance nondestructive evaluation (NDE) capabilities. Preliminary results have shown that a conductive concrete can facilitate the utilization of an eddy current technique, where damages in a conductive specimen were easier to detect compared with a non-conductive substrate. While such results demonstrated the promise of using conductive concrete to facilitate and potentially accelerate the NDE process, the fabrication of an homogeneous conductive concrete is technically or economically challenging, depending on the conductive filler used in the process.

In this paper, we propose a new cementitious composite to accelerate NDE. The composite uses inexpensive carbon black particles (CB) and a block-copolymer. The purpose of the block co-polymer, a styrene-ethylene-butylene-styrene (SEBS), is to facilitate the creation of conductive chains, therefore reducing the necessary concentration of conductive filler required to achieve electrical percolation. Several cementitious composite specimens of various concentrations of CB are fabricated, and results show that the utilization of SEBS reduces the electrical percolation threshold by approximately 50% with a gain on electrical conductivity relative to a non-conductive specimen mix of approximately 33%. Strain-sensing tests also demonstrate that SEBS-based specimens have good sensing properties, but lag behind those of conductive concrete specimens fabricated with CB only.

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INTRODUCTION

Detecting damage in large concrete structures is a difficult task. The vast majority of concrete condition assessments are reported to be conducted by visual inspection, which is highly time consuming, does not look below the surface, and relies on the inspector's judgment. Various NDE techniques can potentially be used in-situ to provide more complete and potentially more accurate information about structural health. Of interest in this paper is the NDE of large-scale cementitious substrates such as pavements and nuclear confinement structures. However, NDE techniques typically used in monitoring such components, including eddy current and ground penetrating radar, are relatively slow processes and given the attenuating nature of concrete, some defects can be very difficult to detect even using NDE technology.

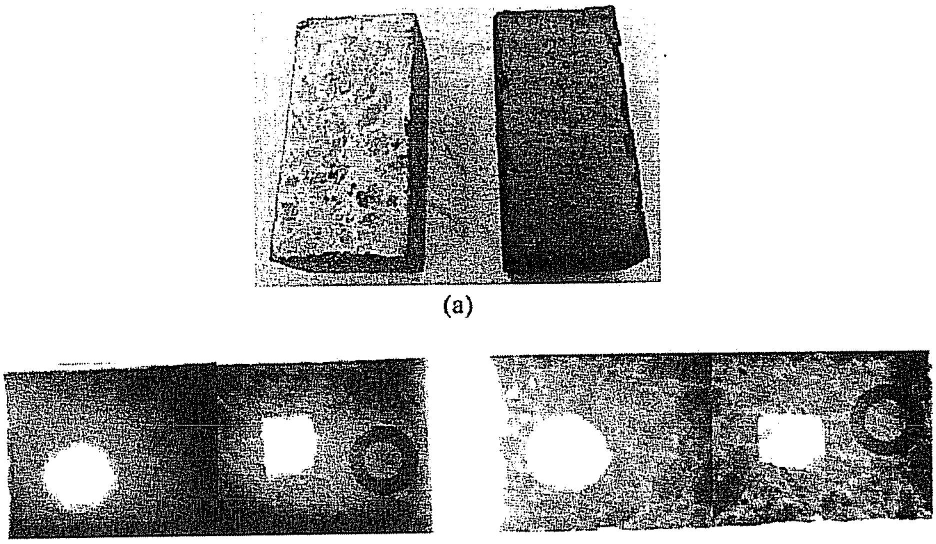
As it will be demonstrated in the next section, it is possible to utilize smart concrete to enhance NDE capabilities. Smart concrete is fabricated by adding conductive particles within the cementitious matrix. Beyond a given concentra- tion of conductive particles, the cementitious material becomes conductive. This concentration is termed percolation threshold. Several studies have analyzed cementitious materials filled with carbon nanoinclusions[1–4] that include carbon black (CB), carbon fibers, and carbon nanotubes (CNTs). The enhanced electrical properties of concrete are leveraged to improve NDE capabilities, in particular for eddy current. While the concept of conductive concrete has been used in pavements primarily for de-icing purposes,[5–7] it is yet to be implemented as a tool to improve fault detection. The development of multifunctional cementitious materials for sensing purposes is more complex. This is mainly because the nanocomposite must provide a linear and accurately measurable signal with respect to a change in the measured state. There are fundamental challenges that limit the cost-effective fabrication of large volumes of conductive cementitious materials, namely:

- There is a trade-off between the cost of the selected conductive filler and its conductivity. Lower-conductivity nanofillers such as carbon black require higher loading, which may significantly affect the strength of the nanocomposite. Conversely, higher-conductivity nanofillers such as CNTs require fewer particles, but result in a much higher fabrication cost;
- The majority of the proposed conductive cementitious materials necessitate a complex fabrication process to ensure homogeneous dispersion of nanoparticles,[3] which impedes scalability. This is a problem particularly associated with CNTs that tend to spontaneously agglomerate in bundles due to Van der Waals attraction forces;[8]

The authors have previously used CB particles to create conductive materials[9] due to the particles' low cost and ease of dispersion.[10] It was found that CB particles are excellent candidates in producing scalable smart materials, such as smart road pavements. However, the required concentration level of CB to achieve electrical conductivity is prohibitive (see Wen & Chung[11] and Li et al.[12] for instance). Such a high doping content may have detrimental effects on material's strength. A solution is to utilize a polymer to facilitate the creation of conductive chains, therefore reducing the amount of required conductive filler. For example, ref.[13] showed that the use of styrene-butadiene-styrene to disperse CB particles resulted in a 40% reduction in the percolation threshold. Ref.[14] studied the percolation in composites where polyethylene was mixed with polystyrene, and found that CB was selectively distributed on the polyethylene phase and thus led to reduced percolation thresholds compared with the utilization of monophase polymers as composites.

In this paper, we study the utilization of a block co-polymer, namely styrene-ethylene/butylene-styrene (SEBS), to accelerate percolation of CB-filled cementitious materials, therefore decreasing the requisite loading of filler for the fabrication of smart cementitious materials that could be used to accelerate NDE of concrete components. The objective is to produce an homogenous conductive mix using a smaller amount of CB than what would be needed for a neat cementitous matrix. This is done by fabricating cementitious specimens termed "SEBS-CB specimens". The study builds on the results of a preliminary investigation presented in ref.15 A more extensive study is presented here to support the hypothesis that SEBS can be used to accelerate percolation of cement paste filled with CB. Also, the fabrication procedure has been updated and validated in order to improve on the dispersion of the SEBS-CB mix. Remark that work presented in this paper focuses on the application of SEBS-CB to create conductive cement paste. The quantification of NDE capabilities using SEBS-based cementitious mixes is left to future work, because the performance of SEBS-CB in concrete is yet to be studied.

The rest of the paper is organized as follows. Section 2 presents preliminary results demonstrating that a smart concrete can be used to improve eddy current capabilities. Section 3 introduces the research methodology, which includes the fabrication process of the conductive mixes. Section 4 presents and discusses results. Section 5 concludes the paper.



(a)

(b) (c)

Figure 1: Neat (left) and conductive (right) specimens (a); and digital radiographs of concrete specimens showing embedded defects: reference (neat) specimen (b); conductive specimen (c).

ENHANCED NDE USING CONDUCTIVE CONCRETE

We preliminarily investigated the utilization of conductive concrete to improve damage detection using eddy current-based NDE. In this work, two concrete specimens of dimensions 203.2 x 101.6 x 88.9 mm^3 (8 x 4 x 3.5 in^3) were poured, one made conductive by adding CB particles, one neat. Both specimens are shown in Fig. 1(a). Identical defects were placed into both specimens during the pouring process. Damages included a ping pong ball, a square piece of foam, and a steel washers, shown in Figs. 1(b)-(c) left, middle, and right, respectively.

A contact sliding probe was used to detect damage. The probe was used in both a pulse-echo and differential modes at a 5 kHz frequency. Figure 2 shows the results from the preliminary investigation. A study of the impedance planes reveals that the eddy current probe positively responded to damages in the case of the specimen doped with CB, while the probe negatively responded to damage in the case of the plain specimen. While results presented in this section are qualitative in nature, they demonstrate that smart concrete could be used to enhance NDE of concrete components.

METHODOLOGY

SEBS-CB mixes are fabricated using SEBS type Mediprene obtained from VTC Elastoteknik AB, Sweden (density = 930 kg/m3), CB type Printex XE-2B (2% ash content and 500 ppm sieve residue 45 m) acquired from Orion Engineered Carbons, TX, Portland cement type I/II locally purchased from Ash Grove Cement Company, and copper meshes used to form the electrodes purchased from McMaster-Carr, IL. All the materials are left non-altered, except for the agglomerated CB particles that were broken down using a ball milling process for a duration of 24 hours. Figure 3 shows pictures of the CB particles as received (Fig. 3(a)) and after ball milling (Fig. 3(b)), in which the CB particles show finer and more homogeneous geometrical shapes. The average particle size decreased from approximately 900 μm to approximately 45 μm, which facilitated their dispersion within the cementitious matrix. SEBS-CB specimens were fabricated and their electrical behaviors compared with specimens fabricated with a cementitious matrix doped with CB only termed "CB-only specimens".

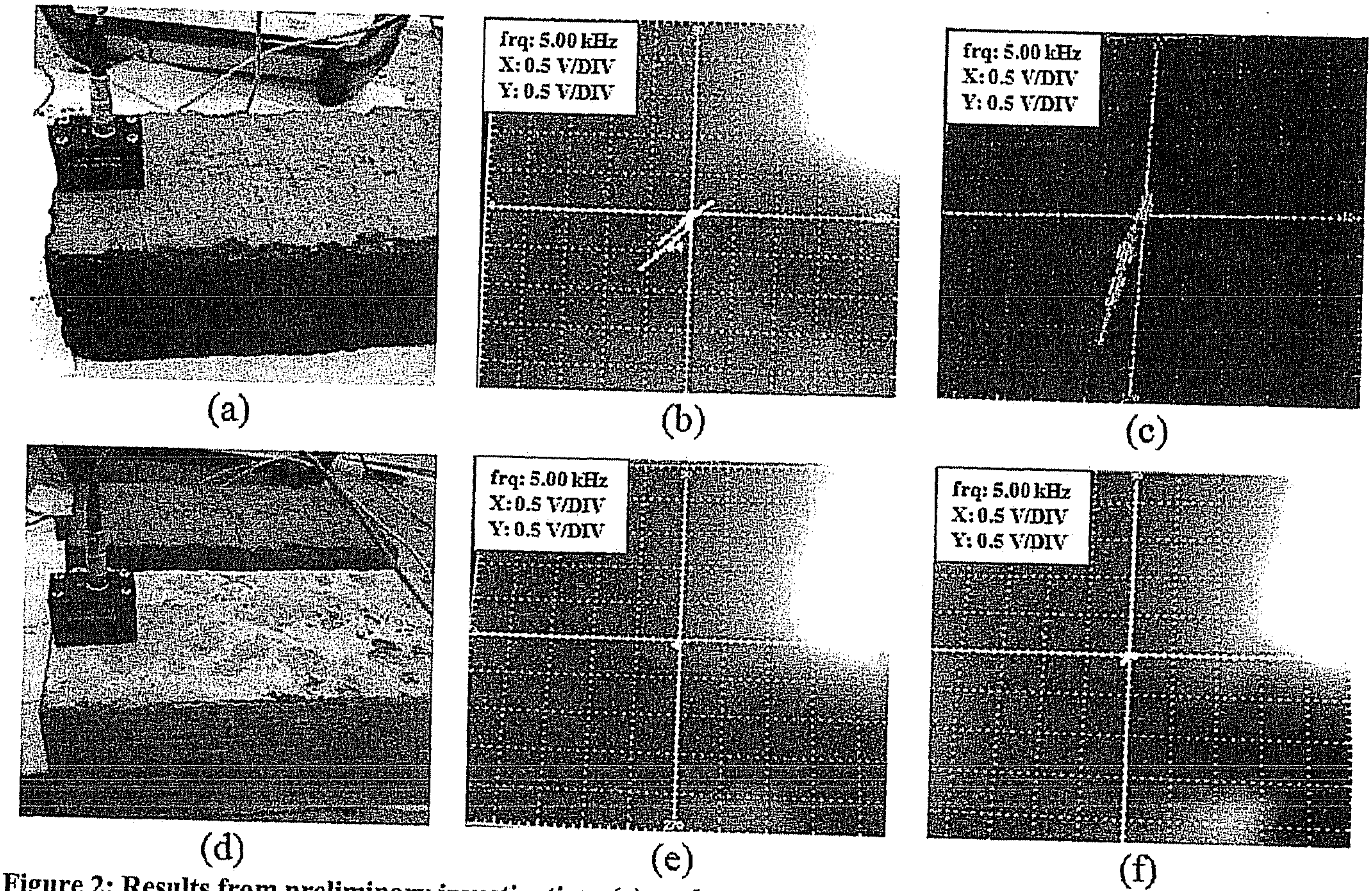


Figure 2: Results from preliminary investigation: (a) probe on conductive specimen; (b) positive response to the ping pong ball (conductive specimen); (c) positive response to the washer (conductive specimen); (d) probe on control specimen; (e) negative response to the ping pong ball (control specimen); (f) negative response to the washer (control specimen).

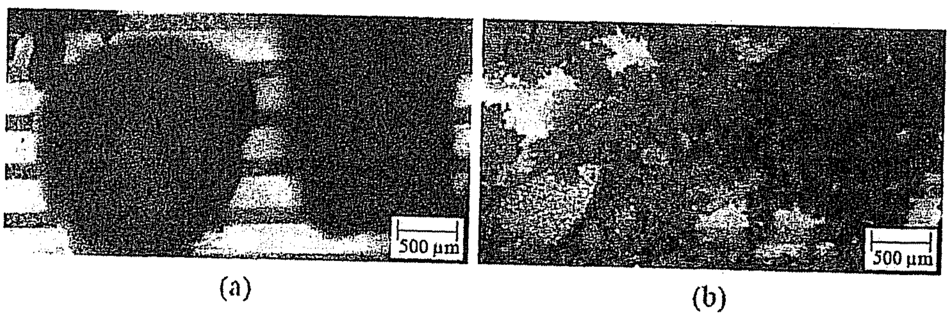


Figure 3: CB particles: (a) before ball milling; and (b) after 24 hours of ball milling.

CB-only specimens

Figure 4 illustrates the fabrication process of a CB-only specimen. The required levels of CB loadings were weighed and mixed in a high shear mixer with the required amount of water needed for a 0.45 water/cement (w/c) ratio. CB particles were mixed in water for 5 minutes in a blender, and cement added to the CB-water solution and mixed for 2 minutes in a Hobart mixer at the highest speed. After, 6 ml of plasticizer were added and hand mixed. Specimens were then cast in molds of dimensions 5.1 x 5.1 x 5.1 cm^3 and vibrated/compacted to eliminate air voids. Two sets of copper mesh electrodes were inserted in each poured specimen. The specimens were covered with a damp cloth allowed to cure overnight before being demolded and placed in a curing room at 100% relative humidity and 22 °C for 7 days. The specimens were then air cured for four days to let water evaporate. Figure 4(6.) is a picture of a completed CB-only specimen.

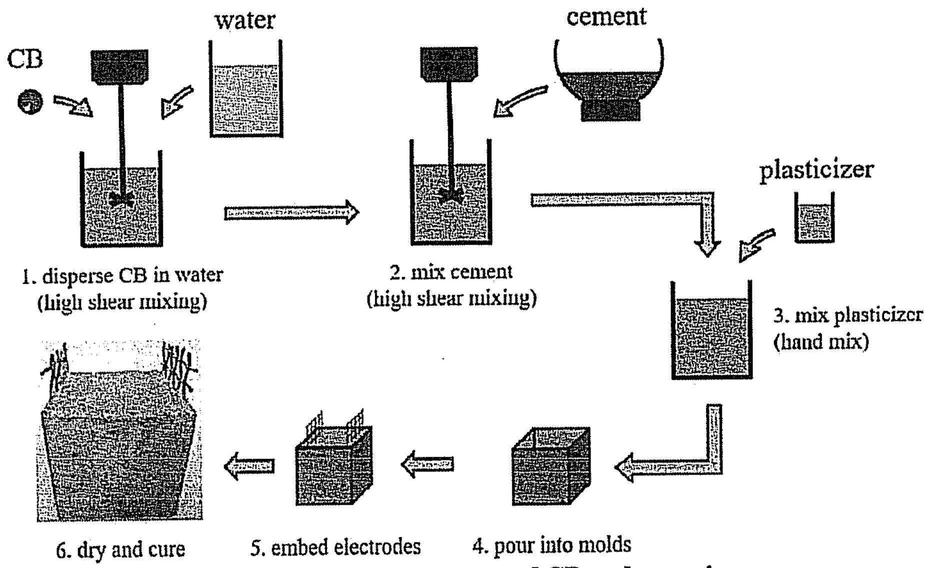


Figure 4: Fabrication process of CB-only specimens.

Table I lists all different CB-only specimen types. Mixes were designed for constant volumes of 164 cm^3 to produce 25% extra material. Three specimens per type were fabricated and tested, for a total of 24 specimens. Remark that while the w/c ratio remained constant for each specimen, additional water was needed due to water absorption by CB particles.

Table I: CB-only specimens

specimen type (#)	water (ml)	CB (g)	cement (g)	%CB (vol%)
1	293	0	651	0
2	292	1.65	649	0.18
3	291	3.30	647	0.36
4	290	5.00	645	0.54
5	289	6.65	643	0.71
6	286	9.00	630	0.96
7	286	12.0	630	1.25
8	286	15.0	630	1.60

SEBS-CB specimens

Figure 5 illustrates the fabrication process of an SEBS-CB specimen. The SEBS polymer was first dissolved in toluene in the ratio of 60 g of SEBS per 500 ml of toluene. The amount of SEBS used in the fabrication of SEBS-CB specimens was governed by the quantity of CB to be added to ensure that the SEBS solution would not be saturated by CB particles. A mass of 3.41 g of SEBS was used for CB loadings up to 0.71 % volume, while a mass of 11.37 g of SEBS was used for CB loadings greater than 0.71 % volume to prevent saturation of CB within the SEBS solution. CB was added to the SEBS solution in the desired loading amount before the mixture was mixed at high shear for 3 minutes. A neat cement paste mix was prepared by combining cement and water at a 0.45 w/c ratio mixed for 2 minutes in a Hobart mixer at the highest speed. After, the SEBS-CB conductive paint was incorporated in the neat cement paste with 0.1 g of sodium lauryl sulphate (SLS) per 131 cm^3 of materials (volume of one specimen), which is a surfactant used to disperse the petroleum-based conductive paint with water, and mixed for 2 additional minutes in the Hobart mixer. The specimens were then poured into molds of dimensions 5.1 x 5.1 x 5.1 cm^3 and vibrated/compacted to eliminate air voids. Two sets of copper mesh electrodes were inserted in each poured specimen. The specimens were covered with a damp cloth and allowed to cure overnight before being demoulded and placed in a curing room at 100% relative humidity and 22 °C for 7 days. The specimens were air cured for four days to develop polymer microstructure and let water evaporate. Figure 5(7.) is a picture of a completed SEBS-CB specimen.

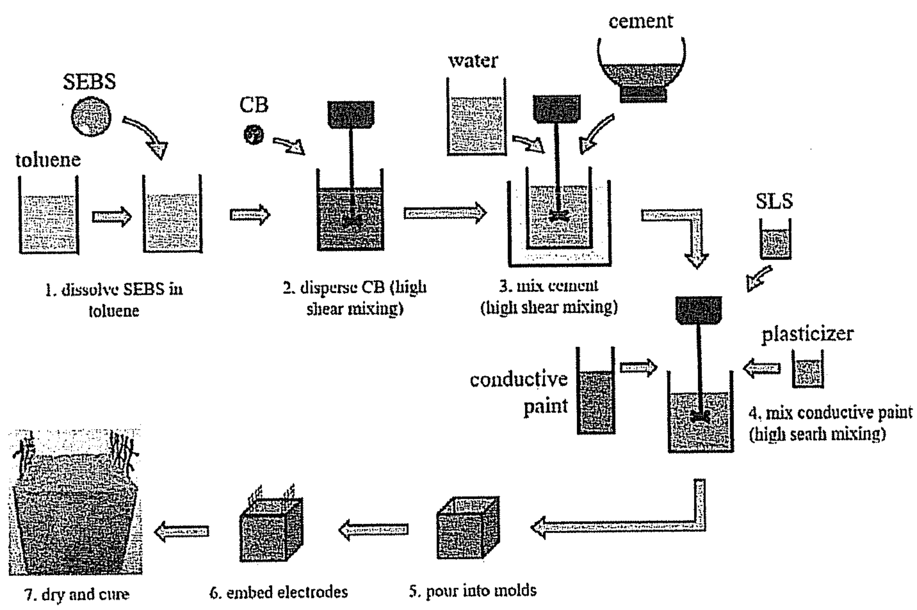


Figure 5: Fabrication process of SEBS-CB specimens.

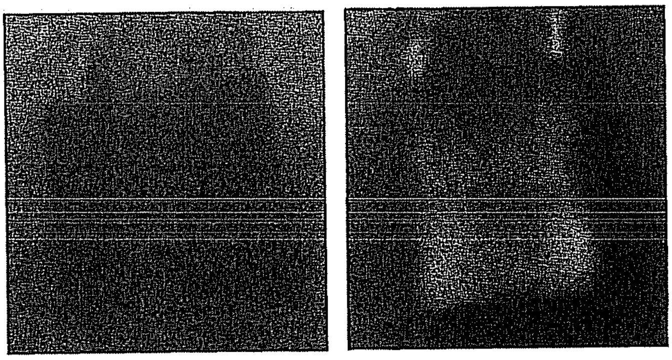
Table II lists all different SEBS-CB specimen types. Three specimens per type were fabricated and tested, for a total of 18 specimens. Only loadings up to 0.96% CB were considered, because it was found that specimens with 1.25% CB loadings and beyond were difficult to fabricate due to the poor workability of the mix, resulting in heterogeneous dispersions.

Table II: SEBS-CB specimens

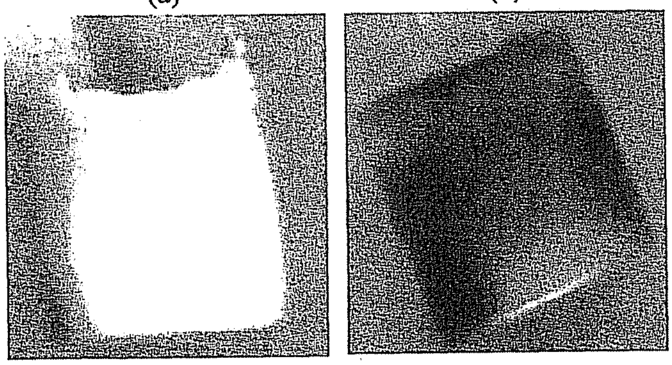
specimen type (#)	water (ml)	CB (g)	SEBS (ml)	cement (g)	%CB (vol%)
1	261	0	15	585.9	0
2	261	1.65	15	585.9	0.18
3	261	3.30	15	585.9	0.36
4	261	5.00	15	585.9	0.54
5	261	6.65	15	585.9	0.71
6	234	9.00	30	519.8	0.96

Measurements

The quality of dispersion in each specimen was inspected using an FLIR A35 thermal camera capturing images at approximately 1 Hz. Specimens were loaded with an AC voltage of 60 V. Thermal images were used to visually assess the distribution of CB particles that appeared in the images as bright pigments. Figure 6 shows thermal images of typical specimens. Figure 6(a) is an electrically unloaded specimen, where the cementitious block shows as a black cube. Figures 6(b) and (c) are examples of well dispersed specimens containing high levels of CB particles. Figure 6(d) shows the example of a badly dispersed specimen for which the CB particles sank and agglomerated at the bottom. Any specimen identified as badly dispersed was discarded and re-fabricated.



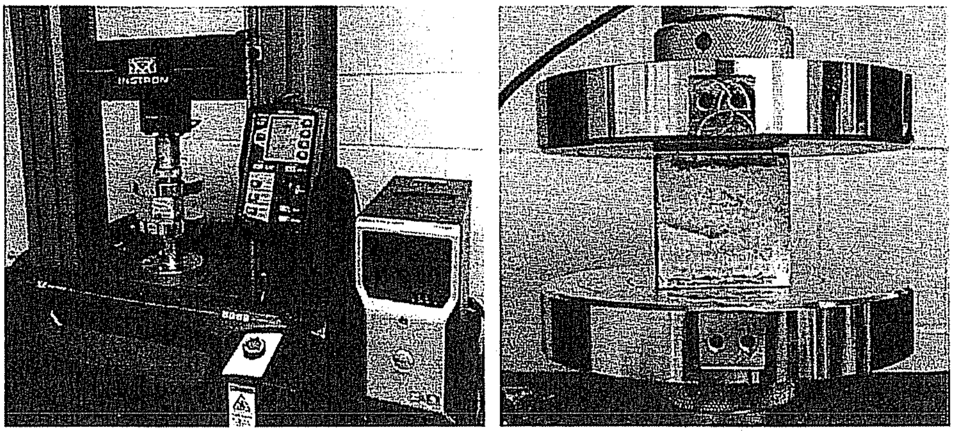
(a) (b)



(c) (d)

Figure 6: Thermal images of (a) electrically unloaded specimen (typical); (b) electrically loaded specimen - 1.60% CB-only; (c) electrically loaded specimen - 0.71% SEBS-CB; and (d) electrically loaded specimen with bad CB dispersion.

The resistance of each specimen was measured to determine the change of resistivity as a function of the CB loading, measured using an LCR bridge at 100 kHz. These measurements were used to obtain the percolation curves.



(a) (b)

Figure 7: Experimental configuration. (a) Cementitious specimen installed in the universal testing machine; and (b) zoom on the cementitious specimens.

The resistance was also measured to determine the specimens' sensitivity with respect to strain to verify and compare the electrical properties of specimens as a function of the CB loading. Strain was applied on each specimen using a universal testing machine type Instron (50 kN capacity), and loaded in steps of 40 μE at a rate of 0.0005 in/sec up to 400 μE, and then unloaded using the same steps and rate. Figure 7 is a picture of the strain test configuration.
The measured sensitivity of the specimens was benchmarked against a theoretical electromechanical model. Con- sider a cementitious specimen with two embedded electrodes of embedded width b and height h, spaced by a distance L, subjected to a uniaxial strain (perpendicular to the electrodes) provoking a change in the electrode distance ΔL. The specimen can be modeled as a resistor of resistance R

$$R = \rho \frac{L}{A} \tag{1}$$

where ρ is the resistivity of the material and $A = b \cdot w$ the cross section area of the embedded electrodes. Assuming small strain, taking the finite difference of Eq. (1) yields

$$\begin{aligned} \frac{\Delta R}{R} &= \frac{\Delta \rho}{\rho} + \frac{\Delta L}{L} - \frac{\Delta A}{A} \\ &= \frac{\Delta \rho}{\rho} + (1 + 2\upsilon)\varepsilon \end{aligned} \tag{2}$$

with

$$\varepsilon = \frac{\Delta L}{L} \tag{3}$$

where υ is the Poisson's ratio of the material and ε the axial strain. Using Eq. (2), an expression for the gauge factor λ can be obtained:

$$\begin{aligned} \lambda &= \frac{\frac{\Delta R}{R}}{\varepsilon} \\ &= (1 + 2\upsilon) + \frac{\frac{\Delta \rho}{\rho}}{\varepsilon} \end{aligned} \tag{4}$$

The first term in Eq. (4) represents the change in resistance due to the change in the specimen's geometry, while the second term represents the piezoresistive effect.

RESULTS AND DISCUSSION

Using the methodology described above, the percolation thresholds and strain sensitivity of the SEBS-CB and CB-only specimens were obtained and compared. Results are presented and discussed in what follows.

Percolation Thresholds

The percolation curve for the SEBS-CB specimens is compared against that of the CB-only specimens in Fig. 8. The SEBS-CB specimens show a change in the electric phase between 0.36% and 0.71% CB, while the change in the electric phase of CB-only specimens occur between 0.71% and 1.25%. It can be concluded that the addition of SEBS resulted in reducing the percolation threshold by approximately 50%. The average resistivity of SEBS-CB specimens drop from approximately 81,000 Ω·cm before percolation to approximately 7,100 Ω·cm after percolation, while the average resistivity of CB-only specimens drop from approximately 21,000 Ω·cm before percolation to approximately 950 Ω·cm after percolation. Thus, percolated SEBS-CB specimens exhibit a resistivity ρ that is approximately 67% of the unpercolated CB-only specimens, but approximately 7.5 times higher than the percolated CB-only specimens. This apparent vertical shift in the percolation curve is due to the insulating nature of the polymer.

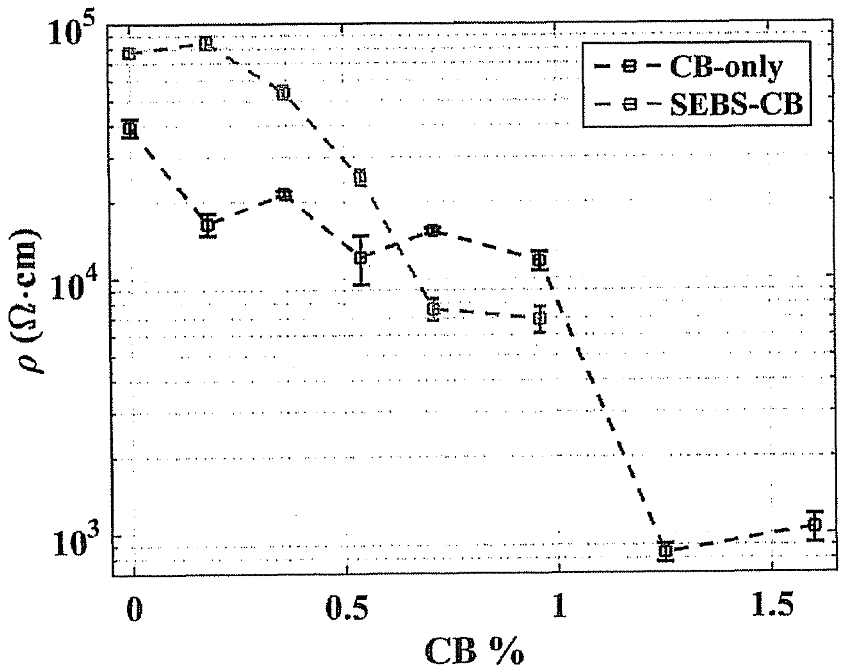


Figure 8: Resistivity ρ versus CB% for CB-only and SEBS-CB specimens.

Strain Sensitivity

The results of the strain sensitivity tests are plotted in Figs. 9 (SEBS-CB specimens) and 10 (CB-only specimens). The slope of the linear fit is the gauge factor λ (Eq. (4)). The gauge factor results are assembled in Table III. The optimal gauge factor for CB-only is obtained at 0.96% CB, while the optimal gauge factor for the SEBS-CB is obtained at

0.54%CB loading. These results agree with those from the percolation threshold study discussed in the last subsection. However, the optimal gauge factor for the SEBS-CB specimens is significantly lower than that of CB-only specimens, which yielded a gauge factor 4.7 times higher (λ = 178 for CB-only versus λ = 38 for SEBS-CB).

A closer inspection of results shows that the strain sensitivity of CB-only specimens exhibit a good linearity, except for the 0.96% loading at low levels of strain. Such nonlinearity can be attributed to the piezoresistive effect being more important at low strain levels around the percolation threshold. On the other hand, the SEBS-CB specimens exhibit higher levels of nonlinearities. This electrical behavior could be partly attributed to the piezoresistive effect, and more importantly to the presence of the polymer that results in an non-negligible capacitance, whereas the assumption yielding Eq. (1) becomes weak. The modeling of the nonlinearities in the SEBS-CB specimens is left to future work.

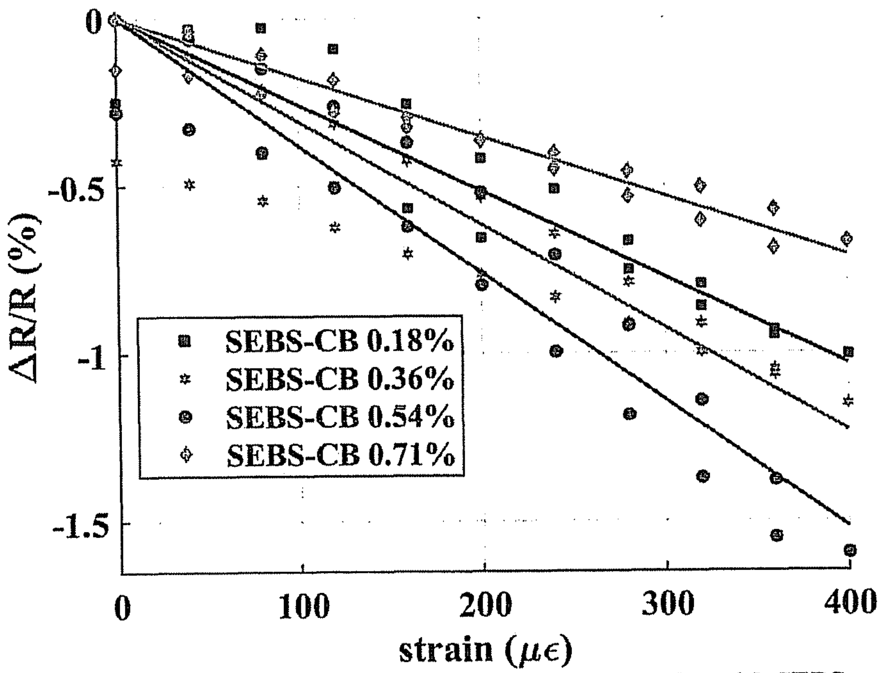


Figure 9: Percent change in resistance v/s CB loading for CB with SEBS specimens

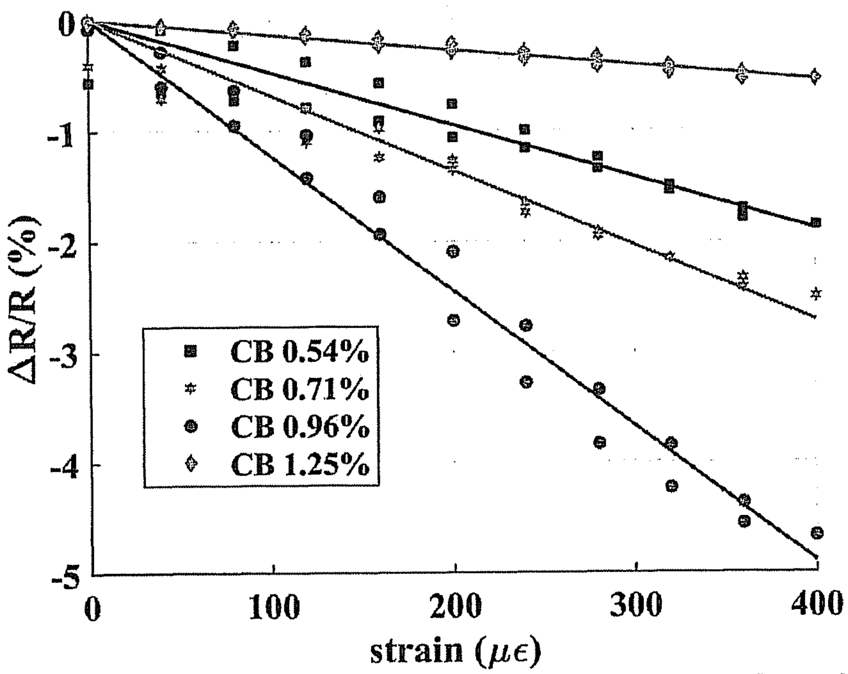


Figure 10: Percent change in resistance v/s CB loading for CB only specimens

Table III: Experimental gauge factors λ

CB%	SEBS-CB	CB-only
0.18%	25.8	–
0.36%	30.8	–
0.54%	38	47.3
0.71%	17.7	82.5
0.96%	–	178
1.25%	–	17

CONCLUSION

In this paper, we have investigated a novel cementitious composite mix that could be used for accelerating nondestructive evaluation of large-scale cementitious components, such as road pavements and nuclear confinement structures. Preliminary results demonstrated that utilizing a smart concrete substantially facilitated eddy current-based inspections, because of the electrically conductive nature of the mix. While such mix utilized carbon black (CB) filler to provide an inexpensive solution that would also be easy to produce at large scales, the amount of CB required for the material to electrically percolate is high and could lead to a decrease in the material's mechanical properties. The authors proposed to utilize a block-copolymer, a styrene ethylene butylene sterene (SEBS), to facilitate the creation of conductive chain in the composite, therefore decreasing the requisite amount of CB necessary to achieve electrical percolation.

Several CB-based specimens were fabricated, some containing SEBS (SEBS-CB specimens), some without (CB- only specimens). The electrical resistance of each specimens was measured, and the resistivity computed and plotted versus the CB loading to obtain percolation curves. A comparison of the percolation curves showed that the utilization of SEBS resulted in a reduction of the percolation threshold by approximately 50%, yet yielding percolated specimens that were 7.5 times more resistive than the percolated CB-only specimens, but with a gain of 67% on the resistivity of unpercolated CB-only specimens. This decrease in electrical performance is attributed to the insulating nature of SEBS. A study on the strain sensitivity of the composites showed that a concentration of 0.54% of carbon black resulted in an optimal gauge factor of 38 for SEBS-CB specimens, while this concentration was 0.86% for CB-only specimens, yet yielding a gauge factor of 178. These results confirmed the location of the percolation thresholds from the study of the percolation curves. However, the optimal gauge factor of the CB-only specimens was higher than that of the SEBS-CB specimens by a factor of 4.7. An investigation of the linearity of the specimens showed that SEBS-CB specimens were significantly nonlinear compared with CB-only. This behavior was attributed to the piezoresistive effect at low levels of strain and the non-negligible participation of the material's capacitance.

Empowering NDE using smart concrete has substantial potential benefits, including 1) the detection of defects lo- cated inside a superstructure that are currently difficult to evaluate using existing NDE techniques; 2) the acceleration of the NDE process by enabling the collection of data at a faster rate; and 3) the possibility to use the smart concrete as a distributed specimen network providing static and/or dynamic feedback. Results from this study demonstrated the promise of SEBS at fabricating such smart concrete using less carbon black particles. While the overall electromechanical properties of SEBS-CB specimens were found inferior to those of CB-only specimens, the utilization of smaller concentrations of carbon black could lead to significantly enhanced mechanical properties. Future work includes the study of such mechanical properties, the performance of SEBS-CB in concrete, and quantifying the enhancement of NDE in SEBS-based conductive concrete.

ACKNOWLEDGMENTS

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